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B1B BKE B307 B501

(56) Documents Cited
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US 4865810 A US 4226669 A US 3850368 A

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Online: EPODOC, PAJ, WPI.

(54) Abstract Title
Centrifugal evaporator with 2-dimensional rotation.

(57) A centrifugal evaporator for concentration of samples contained in tubes 61 comprises a holder 62 for supporting the tubes, a rotor 64, a vacuum chamber 76 and means 63 for causing the holder to perform a cyclic movement relative to the rotor. This rotation is such that the tubes are caused to move from a first position in which one side wall is nearest the rotor axis to a second position in which the opposite side wall is nearest the rotor axis. The rotation may be parallel or orthogonal (Fig. 5) to the longitudinal axes of the tubes. In operation, the rotation of the holder 62 mounted on the shaft 63, in combination with rotation about the main shaft 71, causes the liquid sample in each tube 61 to be swept around the inside walls of the tube, thereby increasing the exposed surface area and improving evaporation. In one embodiment the centrifuge has a rotatable drive shaft 63 connected to the holder. In an alternative arrangement (Fig. 5) the holder is pivoted (14, Fig. 5) relative to the rotor (16, Fig. 5) and the pivot point 14 is moved rapidly up and down, so that the liquid sample is alternatively slushed from one side wall to the other.

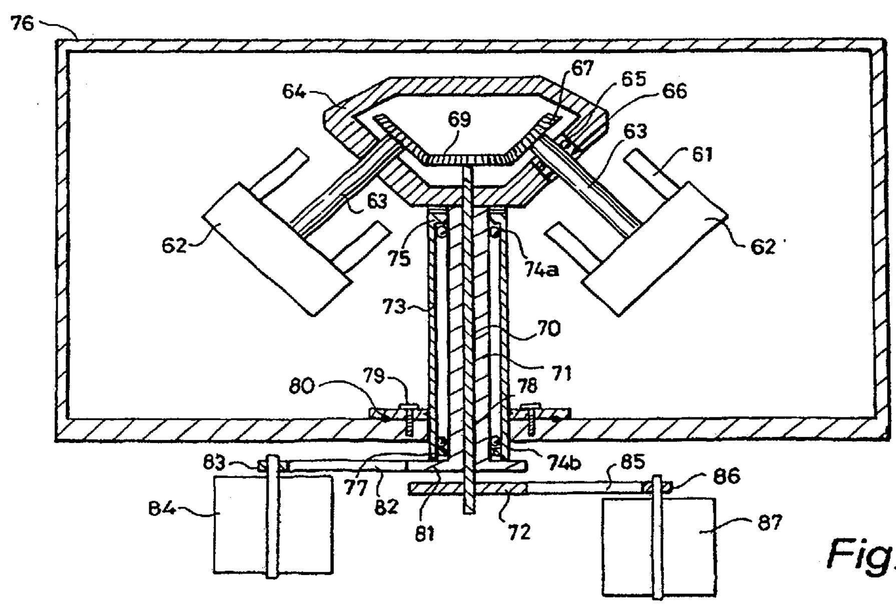

Fig. 6

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At least one drawing originally filed was informal and the print reproduced here is taken from a later filed formal copy.

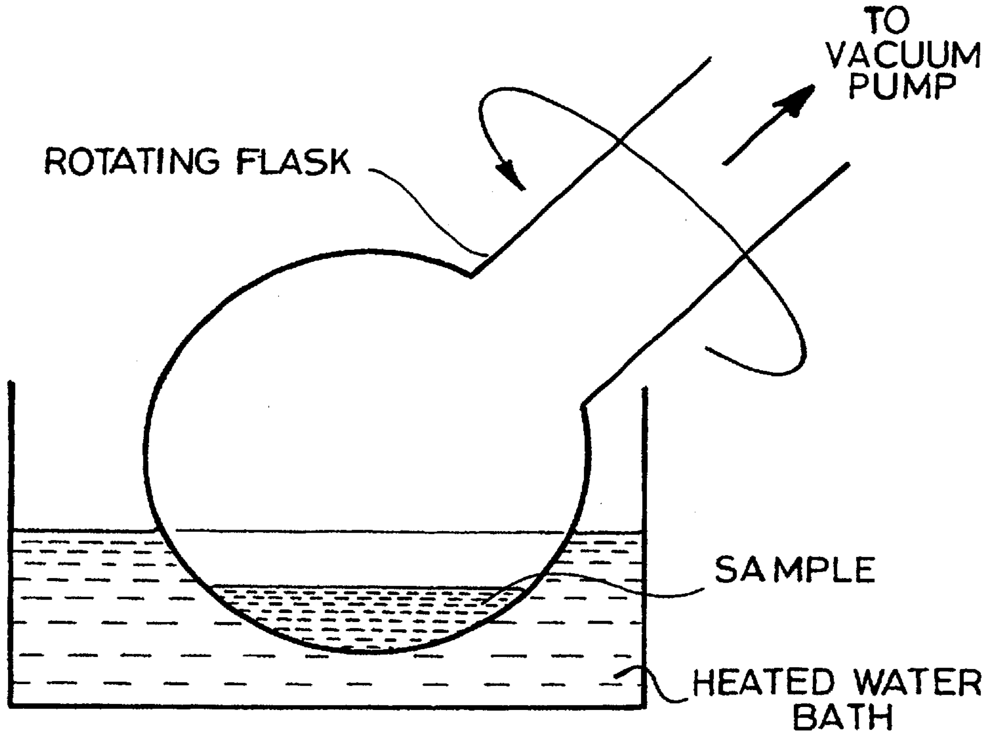

TO
VACUUM
PUMP
ROTATING FLASK
SAMPLE
HEATED WATER
BATH

Fig. 1a

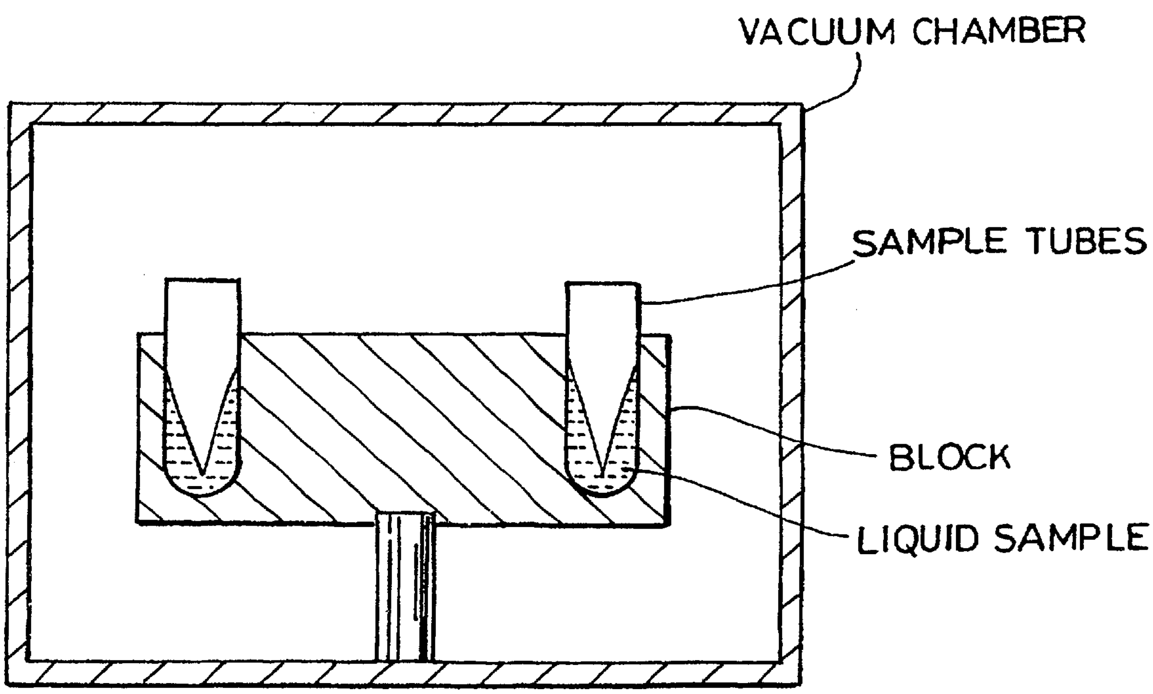

VACUUM CHAMBER
SAMPLE TUBES
BLOCK
LIQUID SAMPLE

Fig. 1b

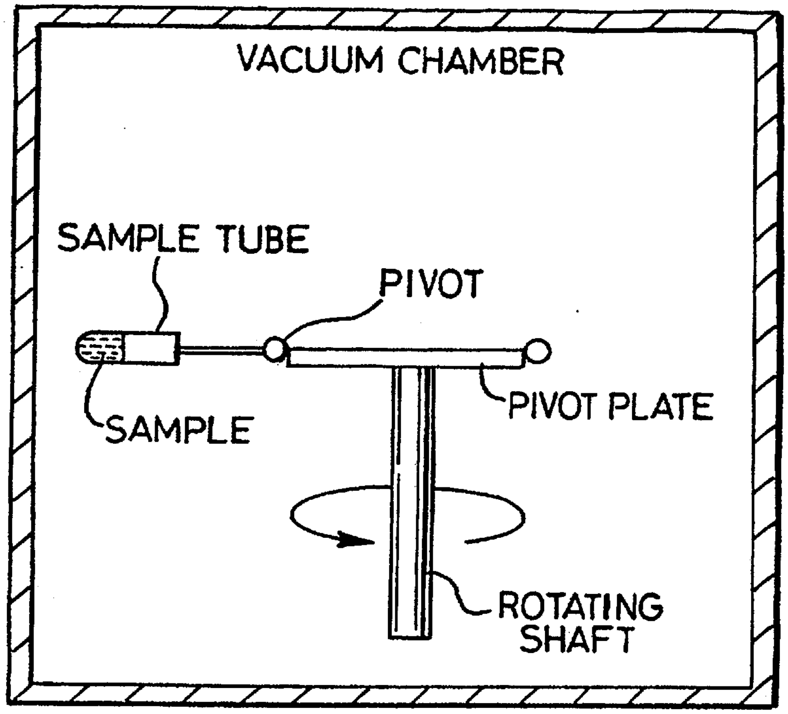

VACUUM CHAMBER
SAMPLE TUBE
PIVOT
SAMPLE
PIVOT PLATE
ROTATING SHAFT

Fig. 2a

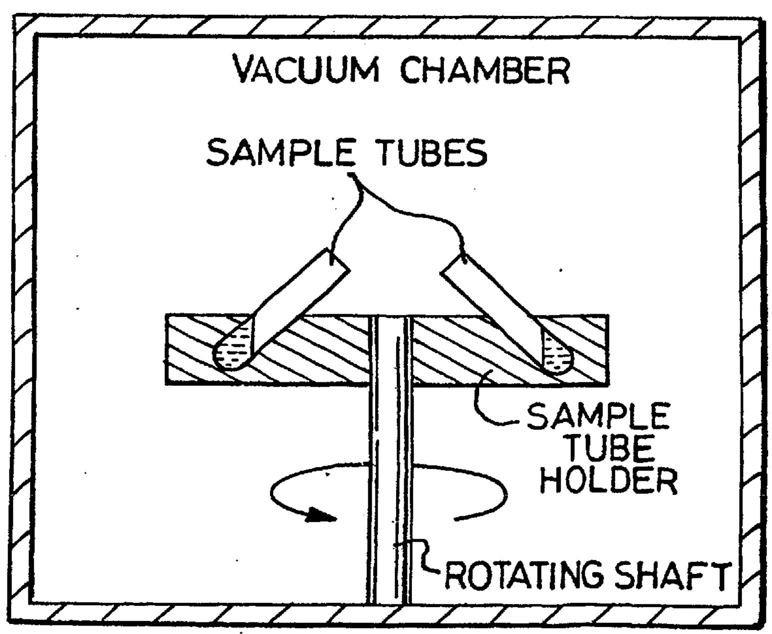
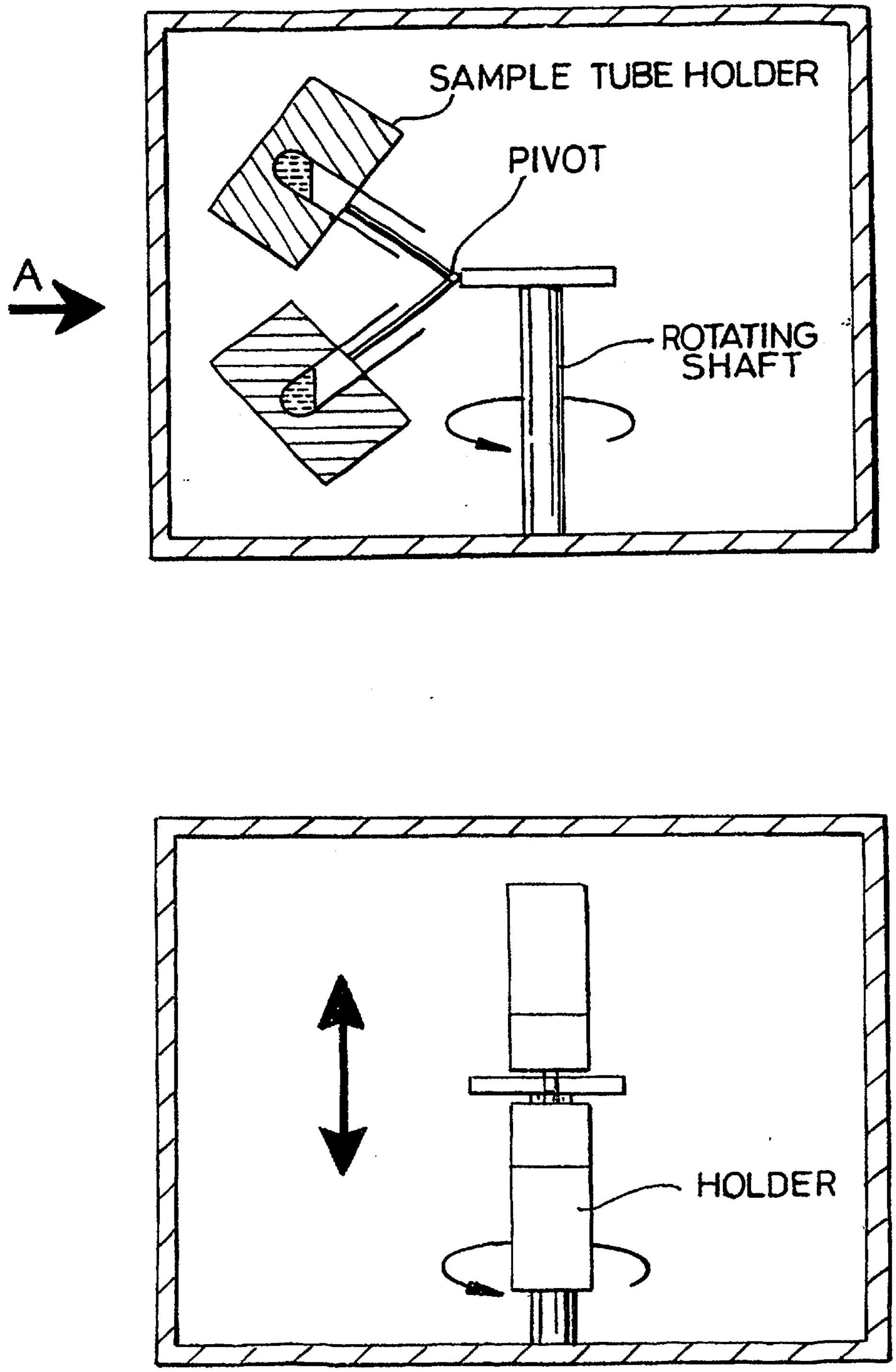

VACUUM CHAMBER
SAMPLE TUBES
SAMPLE TUBE HOLDER
ROTATING SHAFT

Fig. 2b



View from A

Fig. 3

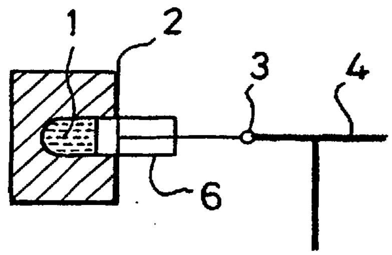


Fig. 4a

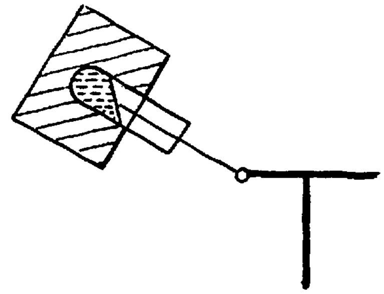


Fig. 4b

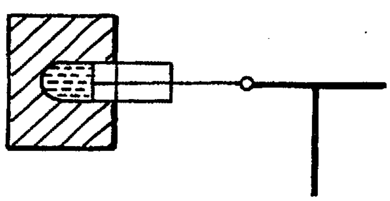


Fig. 4c

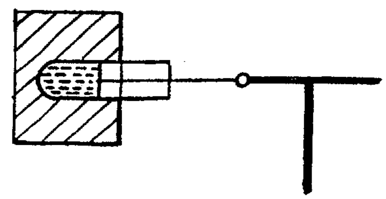


Fig. 4d

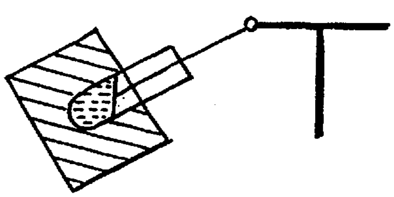


Fig. 4e

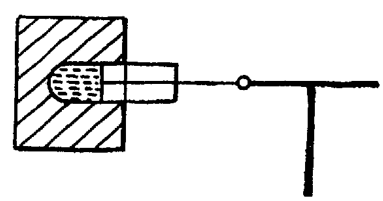


Fig. 4f

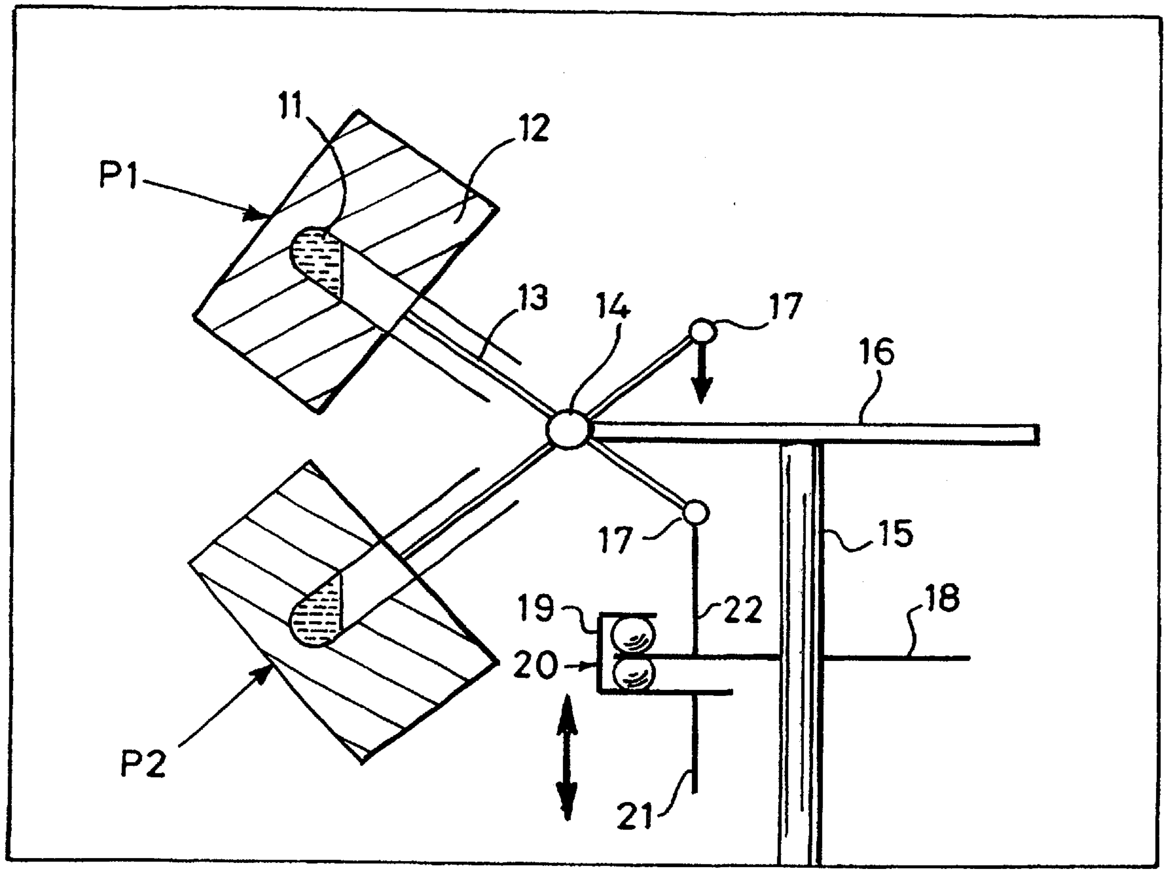


Fig. 5

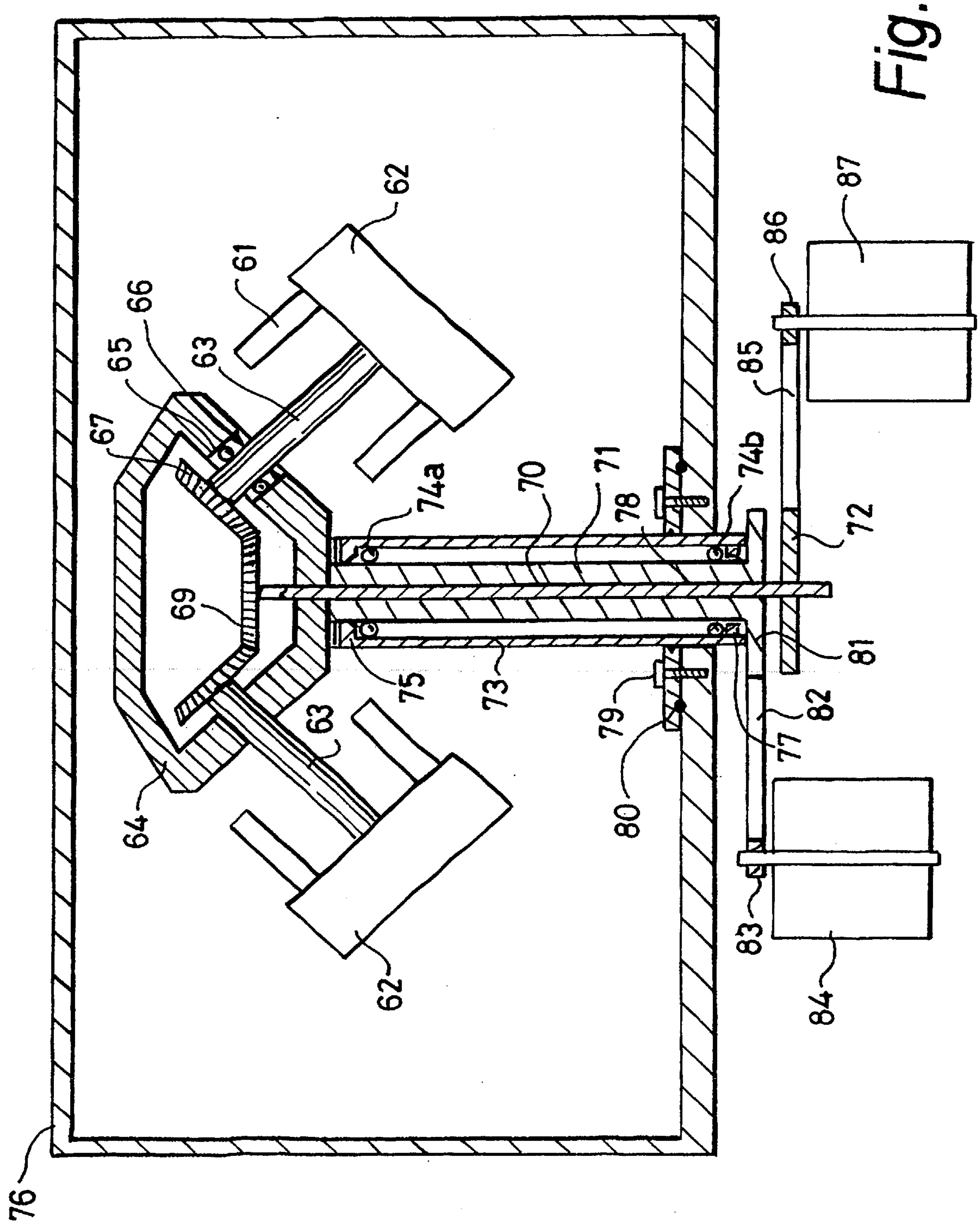

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Fig. 6

Title: Centrifugal Evaporator

Field of invention

This invention concerns centrifugal evaporators.

Background to the Invention

One of the disadvantages of centrifugal evaporators compared with some other forms of evaporators such as rotary evaporators or vortex evaporators is that they evaporate liquids more slowly. This is in more measure due to the lack of agitation of the liquid in such machines.

Thus in rotary evaporators a flask containing a liquid sample is rotated about an inclined axis as illustrated in Figure 1a so that the sample liquid is continuously dragged up by the rotating flask into a thin film covering the whole diameter of that part of the flask in which the liquid lies.

In vortex evaporators (Figure 1b) sample tubes are held in a block to which orbital motion is imparted, and thus also to the sample tubes, which causes the liquid in each of them to form a vortex in the tube, as illustrated.

Both of these forms of agitation cause the liquid to spread out over a large area in the sample container. This results in the advantage of a large surface area for coupling heat into the liquid from its container and speeding evaporation.

Another advantage of rotary and vortex evaporators is that the liquid is continuously mixed so that the evaporating surface is continually refreshed, and compositional variations in the bulk of the evaporating liquid are avoided. This means that the bulk liquid has substantially the same composition as that in the surface and will be maintained at

substantially the same temperature.

In contrast, in centrifugal evaporators, illustrated in Figures 2a and 2b, there is no tendency for continued movement between the bulk of the liquid and the surface, and as evaporation takes place, the evaporating surface becomes cooler than the bulk of the liquid, and preferential evaporation of more volatile components from the evaporating surface of mixed liquids leads to compositional variation through the bulk of the liquid between the liquid in the region surface and the remaining bulk of the liquid. Both these effects lead to increased probability of sample loss due to spitting of liquid out of the tube. This is caused by local superheating of the bulk liquid below the surface liquid layer, as the pressure is lowered to a value which allows evaporation of the surface layer of liquid. Such local superheating arises because the pressure is lowered below that at which the liquid boils at the temperature of the bulk liquid. Regions of the superheated bulk liquid then erupt explosively when a nucleus is formed, and enough vapour is generated instantly, to remove the local superheating. The result is to propel sample liquid out of the tube.

On the other hand rotary and vortex evaporators suffer from at least one disadvantage, when compared with centrifugal evaporators, namely that the evaporation pressure must be manually controlled, not only to allow evaporation to take place, but also to avoid boiling of the liquid which, in the absence of applied other forces, such as centrifugal force in the case of a centrifugal evaporator, would cause liquid to be ejected from the sample tube.

Object of the invention

The present invention seeks to provide an apparatus and method by which the liquid in a tube, well or other container undergoing centrifugal evaporation, is caused to be agitated in such a manner as to increase the surface area of contact between the liquid and its container and to provide continuous replacement of the evaporating liquid surface layer with liquid from regions below the surface layer, in order to achieve the advantages enjoyed by the other evaporation methods mentioned above (and in particular to realise

faster evaporation rates) whilst at the same time retaining the advantage of spitting suppression by the application of a sufficient centrifugal force.

The invention also seeks to provide for improved mixing in a sample liquid, in order to reduce local superheating in the bulk of the liquid below the surface layers and compositional variation of the bulk liquid, thereby reducing still further the tendency for liquid to be ejected, by spitting, from the tube.

Typically, a centrifugal evaporator includes multiple sample tubes (which expression can mean other forms of sample holder such as vials or wells sunk into solid blocks) which are held in a holder and spun with the sample tube axes in a fixed relationship to the axis of rotation of the evaporator, either normal to the axis of rotation of a sample tube holding rotor (hereinafter referred to as the rotor axis) as illustrated in Figure 2a, or at a fixed angle not equal to 90° as shown in Figure 2b.

For clarity only one tube is shown in Figure 2a where there would be at least one more to balance the system. This convention is repeated in other Figures of the drawings.

<u>Summary of the invention</u>

According to one aspect of the present invention there is provided a method of performing centripetal evaporation of a sample contained in a tube having walls and supported in a holder, comprising the steps of mounting the holder on a rotor which is rotatable about a vertical axis within a vacuum chamber, rotating the rotor about its axis with the tube inclined relative thereto, and simultaneously causing the holder to perform a cyclic movement relative to the rotor, such that the tube moves from a first position with one wall nearest the rotor axis to a second position with the opposite wall nearest the rotor axis.

According to another aspect of the invention there is provided a centrifugal evaporator comprising a holder supporting at least one sample tube and mounted on a rotor within a vacuum chamber; first drive means for rotating the rotor about its vertical axis; mounting

means for movably mounting the holder so that the tube is inclined relative to the rotor axis; and second drive means for causing the holder to perform a cyclic movement about the mounting means; whereby the tube is caused to move from a first position in which one wall portion thereof is nearest to the rotor axis to a second position in which the opposite wall portion is nearest the rotor axis.

The mounting means may comprise a plate secured to the rotor and an arm secured to the holder and connected to the plate by a pivot, enabling the holder to move from a first position in which the axis of the tube is at an acute angle to the rotor axis to a second position which the axis of the tube is at an obtuse angle to the rotor axis.

Alternatively the holder may be secured to a drive shaft rotatably mounted from the rotor, the second drive mean causing rotation of the drive shaft.

The invention thus provides practical means for rotating such multiple sample tubes about their own separate axes, or subjecting them to oscillatory motion, in addition to subjecting them to rotation about the rotor axis for achieving the desired increased contact area with the sample tubes and mixing of the sample liquid.

In one method for achieving the desired agitation proposed by the invention, the angle at which the axis of each tube is held, relative to the rotor axis of rotation, is changed while they are rotating.

Normally the tubes are held with their axes at a fixed angle to the axis of rotation. The liquid always quickly adjusts its position under the influence of the centrifugal force so that it is as far away as possible from the rotor axis, and so that its free surface is substantially parallel to the rotor axis. The free surface therefore occupies the position in the tubes as is illustrated in Figure 2.

If the orientation of the tube axis is changed, the liquid will rapidly adjust its position relative to the tube, so that the liquid is again positioned as far away as possible from the rotor axis. This means that the liquid will move to a different position in the tube, thereby

extracting heat from a different part of the tube, and the free surface will consist of different molecules. Some mixing of the liquid will also occur.

If during rotation the tube axis angle is continually changed in this way, the liquid will be spread over a larger area of the tube than it would if the orientation remained fixed (as in Figure 2).

Thus if, in accordance with the invention, in addition to rotation about the rotor axis, the inclined tubes are also continuously rotated about another axis which intersects the rotor in a manner which generates a cone, the liquid in the tubes will be swept around the inside of the tubes to simulate a vortex. Thus the axis of the cone will itself be rotated, which will cause the liquid in each tube to be agitated, to increase its free surface area and extract more heat from the walls of the tube, thereby considerably increasing the rate of evaporation.

Whilst rotation of the tubes in such a conical manner is probably the most effective method of achieving the required result, good results can also be obtained by movements which may be easier to achieve.

The simplest movement is probably to change the inclination of the tube axis in a single (rotating) plane as illustrated in Figure 3. This will move the liquid up and down along opposite side walls of the tube, rather than all the way round the tube, such as is achieved by a conical movement such as described above.

There are several possible and practicable methods of achieving movements of this type including the following examples.

Examples of mechanisms to achieve tube axis movement

Referring to Figures 4, liquid samples 1 are held in tubes 6 in swings or buckets 2 that pivot about pins 3 in a rotating plate or rotor 4. When the rotor is rotating the buckets in a fixed perpendicular plane, the liquid in each tube will take up the position illustrated

in Figure 4(a). When the plane of the rotating plate is suddenly displaced downwards vertically (ie in an axial direction) the buckets will adjust their rotational plane correspondingly to the position illustrated in (c), but not immediately because of their inertia. Thus they will momentarily assume position (b) before moving to position (c). The liquid in the sample tubes 6 will adjust more rapidly to the change and will temporarily move towards the lower wall of the tube as illustrated in position (b). The rotation plane is then rapidly displaced upwards back to its original position, causing the liquid in the tubes momentarily to assume position (e) before returning to position (f), which is the same as the original position (a).

The sample rotation plane can be moved by a variety of means, including moving the entire apparatus, moving the drive shaft on which the rotor rotates, or moving the rotor relative to the drive shaft.

Changing the angle at which the buckets, and therefore the sample tubes, are inclined to the rotation plane can produce the same effect. A way to achieve this is illustrated in Figure 5. The samples 11 are held in tubes in buckets 12 which are held on holding arms 13 which pass through pivots 14 on the outside of plate 16 rotating about a central shaft 15. The inner ends of the holding arms 13 not attached to the buckets are attached through a pivot 17 to actuator rods 22 which are themselves attached to a rotating ring 18 mounted on an axial sliding bearing (now shown) on central shaft 15. An actuator rod 21 is attached to the outer race 19 of a rotary bearing 20. This allows free relative rotation between the rotating and stationary race but couples them in an axial direction. In this arrangement vertical movements of the actuator rod 21 will change the position of the sample bucket from position P1 to position P2 and back. If this is done repeatedly the desired movement of the liquid relative to the tube will be achieved as illustrated and the effect will be similar to that described above caused by moving the sample rotation plane.

The mechanisms described above achieve movement of the tube holder axes in one plane, which also contains the axis of rotation. If the tube axes are simultaneously moved in a plane that is normal to the axis of rotation the tube axes can in fact be made to move in a conical manner, as referred to above, and better results can be obtained.

One method for achieving this movement is to hold the sample tube holder so that the tube axes are inclined at an angle of about 45° to the axis of rotation and rotate the holder about its own axis. This action will cause the tube in the centre of the holder to rotate about its own axis and the axes of the outer tubes will rotate in a manner which describes a cone: they will also be displaced from their original position. This motion will cause the liquid in all the tubes held in the holders to move round the tube thereby increasing free surface area and contact with the tubes. This leads to increased evaporation rates and a lower tendency of a sample liquid to spit out of the sample tubes. The angle at which the tubes are held can be varied from, for example, 35 to 55 degrees. Angles outside this range are also possible but require tubes to be less fully filled to avoid liquid spillage.

One possible embodiment of an evaporator based on this principle is illustrated in Figure 6.

Sample tubes 61 are held within a surrounding vacuum chamber 76 in one or more holders 62 attached to sample holder shafts 63 which are held in a rotating gearbox 64 and which are each mounted in a bearing 65. The bearing contains a means (not illustrated) of locating the shaft 63 axially and a seal 66 to seal the inside of the gear box 64 from the space outside the gearbox. Each shaft 63 has a bevel gear 67 which is attached to the end inside the gearbox, and the gear 67 meshes with another bevel gear 69 which is attached to a spindle 70. The spindle passes down through the centre of a main drive shaft 71 through bearings (not shown) to a pulley 72, which is driven via a belt 85 and smaller pulley 86 by a motor 87. The drive shaft 71 is connected to the gearbox 64 by screws or other means (not shown), and is supported in a drive support 73 by means of upper and lower bearings 74a and 74b. A seal 75 above the upper bearing 74a seals the drive shaft 71 from the vacuum chamber 76, and a shoulder 77 below the lower bearing 74b and a circlip 78 above the bearing 74b axially locates the shaft 71.

The drive support 73 is held in the chamber by means of screws 79, and an O-ring seal 80 seals the inside of the drive support from the vacuum chamber 76.

The drive shaft 71 is attached to a pulley 81, which is driven via a belt 82 and a smaller

pulley 83 by a motor 84.

In operation, samples containing the non-volatile substance, and a volatile liquid in which it is dissolved or otherwise mixed, are placed in the tubes 1 which are placed in holes in the holder 62 or, alternatively, the liquid sample is placed directly in holes in the holder 62. The whole gearbox assembly 64 is then made to rotate, by applying suitable power to the motor 84, at a speed which will provide enough centrifugal force to the samples to prevent sample loss from the tubes when the chamber 76 is evacuated. Power is also applied simultaneously to the motor 87, which will cause the samples to rotate about the axes of the sample holder shafts 63. The rotation of the gearbox will normally be at a speed which will apply 50 - 200g centrifugal force to the samples at say 200-750 rpm, depending on the size of the equipment. Rotation of the samples about the sample holder axis will normally be at a much lower rate say 25 to 150 rpm and controlled by setting the relative speeds of motors 84 and 87. When the samples are rotating at a sufficient speed to prevent sample loss from the tubes, the chamber is evacuated to a pressure that will cause evaporation of the sample liquid. It may also be necessary to apply heat by means that are well known in centrifugal evaporators.

In the embodiment illustrated above two separate motors are used to drive the rotation of the gearbox and the gear system to rotate the sample holders respectively. In practice an arrangement using gears, or other mechanical means, may be used to couple the two drives together. In some arrangements it may even be possible merely to hold the spindle 70 stationary, so causing rotation of the gears 67, and thus the sample holders, in the manner of an epicyclic gearbox.

Clearly it is necessary to provide a means of opening and closing the vacuum chamber 76 to gain access to the samples, together with means for sealing such access and to ensure a vacuum tight chamber and means for applying vacuum to the chamber and subsequently admitting air. However, these are all well-known features in centrifugal evaporators and are not illustrated or described here.

Although the invention (and the various illustrated embodiments) has been described in

relation to centrifugal rotation of the sample tube holders in combination with rotation or orbital motion of the individual holders, the invention is not limited to unidirectional rotation of the individual tubes, and advantageously, the tubes may instead be subjected to an oscillatory rotational motion, so that the tubes do not continue to rotate in one direction. Thus, for example, in the Figure 6 embodiment the motor 87 may be a reciprocating drive. This simplifies the making of electrical connections to sensors in or on the tubes, and avoids the need for slip-ring connections, eg between the gearbox 64 and the tubes 61, since flexible electrical cables can accommodate the oscillatory motion.

Slip-ring connections, or a radio link, will in general be necessary to complete the signal path (and/or supply of power) to sensors (not shown) in or on the tubes, as between the shaft 71 and contacts on the stationary vacuum chamber 76 (Figure 6), or to an external receiver which may be inside or outside the chamber 76.

Claims

1. A method of performing centrifugal evaporation of a sample contained in a tube or well having wall portions and supported in a holder, comprising the steps of mounting the holder on a rotor which is rotatable about a vertical axis within a vacuum chamber, rotating the rotor about its axis with the tube incline relative thereto, and simultaneously causing the holder to perform a cyclic movement relative to the rotor, such that the tube moves from a first position with one wall portion nearest the rotor axis to a second position with the opposite wall portion nearest the rotor axis.

2. A method according to claim 1 in which the cyclic movement comprises pivoting the holder about a pivot point relative to the rotor to enable the tube to be swung from an acute angle to an obtuse angle relative to the rotor axis.

3. A method according to claim 2 comprising the step of moving the pivot point vertically in rapid up and down movements.

4. A method according to claim 1 in which the cyclic movement comprises rotating the holder about an axis parallel to the tube, the speed of rotation being lower than that of the rotor.

5. A method according to claim 4 in which the rotation of the holder is oscillatory and causes the holder alternately to rotate through a minimum angle of approximately 180°.

6. A centrifugal evaporator comprising a holder supporting at least one sample tube and mounted on a rotor within a vacuum chamber; first drive means for rotating the rotor about its vertical axis; mounting means for movably mounting the holder so that the tube is inclined relative to the rotor axis; and second drive means for causing the holder to perform a cyclic movement about the mounting means; whereby the tube is caused to move from a first position in which one wall portion thereof is nearest to the rotor axis to a second position in which the opposite wall portion is nearest the rotor axis.

7. An evaporator according to claim 6 in which the mounting means comprises a plate secured to the rotor and an arm secured to the holder and connected to the plate by a pivot, enabling the holder to move from a first position in which the axis of the tube is at an acute angle to the rotor axis to a second position which the axis of the tube is at an obtuse angle to the rotor axis.

8. An evaporator according to claim 7 in which the plate is movable axially with respect to the rotor axis, and in which the second drive means is arranged to move the plate in rapid up and down movements.

9. An evaporator according to claim 6 in which the holder is secured to a drive shaft rotatably mounted from the rotor, the second drive means causing rotation of the drive shaft.

10. An evaporator according to claim 9 in which the second drive means comprises a driven spindle passing through the centre of the rotor and having a bevel gear engageable with a corresponding bevel gear on the drive shaft.

11. An evaporator according to claim 10 in which the drive to the spindle is oscillatory and causes the holder alternately to rotate through a minimum angle of approximately 180°.

12. An evaporator according to any one of claims 9 to 11 in which the first and second drive means each comprises a pulley wheel driven by a motor via a belt.

13. A method of performing centrifugal evaporation of a sample contained in a tube substantially as herein described with reference to, and as shown in, Figures 4 to 7 of the accompanying drawings.

14. A centrifugal evaporator substantially as herein described with reference to, and as shown in, Figures 3 to 6 of the accompanying drawings.

The Patent Office

INVESTOR IN PEOPLE

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Application No: GB 9929898.6
Claims searched: 1 to 14

Examiner: Matthew Jefferson
Date of search: 21 August 2000

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.R): B1B (BKE); B2P (P10A).

Int Cl (Ed.7): B01D 3/08; B04B 5/04, 9/08; G01N 1/40.

Other: Online: EPODOC, PAJ, WPI.

Documents considered to be relevant:

Category	Identity of document and relevant passage		Relevant to claims
Y	GB 2230203 A	(JOUAN) See abstract and figures.	1, 4 & 6.
A	EP 0838265 A	(BECTON DICKINSON CO.) See abstract and figures.	1 & 6.
A	US 4865810 A	(SIMON) See abstract and figures.	1 & 6.
Y	US 4226669 A	(VILARDI) See abstract and figures.	1, 4 & 6.
A	US 3850368 A	(BOECKELER) See abstract and figures.	1 & 5.
Y	FR 2508815 A	(LAVAUX) See abstract and figures.	1, 4 & 6.

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